Design of Solid State Array for Simultaneous Potentiometric and Impedance Sensing in Gas Phase


Abstract
A general sensing platform consisting of eight modules has been designed and fabricated in silicon. The operating function of this platform has been divided between a chemical sensing chip (CSC) and an electronic service chip (ESC). The CSC uses a gold metallization and a high temperature silicon nitride as the passivation layer. The smallest feature size on the CSC is 20 μm. It houses eight sensing modules each consisting of a pair of gold electrodes for measurement of impedance of the conducting polymer and a field-effect transistor for measurement of chemical modulation of the work function of the same polymer. The modules are separated from each other by a patterned 25 μm thick polyimide. The ESC was fabricated by a standard CMOS technology with 1.25 μm feature size. The CSC and ESC chips are connected by flip-chip bonding which greatly simplifies the packaging. The operation of the combined impedance and work function sensing has been verified by exposure to ammonia in ppm range.

Keywords: Eight solid state sensor array, Impedance sensing, Gas phase, Ammonia

1. Introduction

The ability of a sensor to respond to only one species in the presence of other species is called selectivity. It is clearly a highly desirable performance parameter for any chemical sensor and the quest for achieving this property has occupied researchers in the chemical sensor field for several decades. First of all it is important to realize that chemical sensing is only a part of the process called chemical intelligence gathering [1]. In this process, a single chemical sensor provides the raw data which is processed through some chemometric algorithm to yield information. With the introduction of high-rate data acquisition and processing capacity the strategy for attaining high selectivity of information from chemical sensors has changed. A group of chemical sensors of the same type is called a sensor array. If several different transduction principles are used to interrogate the same chemically sensitive layer (e.g., mass and optical sensing) we talk about higher order sensing [2]. The efficiency of the higher order sensing array in this process is determined by the orthogonality of the responses to different chemical species. The concept of orthogonality can be explained simply by considering an array of many identical glass electrodes. They would be completely nonorthogonal (or colinear). Although the redundancy of such an arrangement could have some advantage, the overall information content obtained from such an array would be nearly the same as that obtained from a single glass electrode.

In this article we describe the concept, design, fabrication, and evaluation of a higher order chemical microsensor array, GT01 (standing for Georgia Tech, series 01) which is intended to become a platform for a broad range of gas sensing needs. It consists of eight sensing modules (i.e., an 8-sensor array) and employs two transduction principles: chemical modulation of work function (WF) of sensing layers, and chemical modulation of impedance of the same sensing layers. It is, therefore, a first order chemical sensor (a zero order sensor would measure only one of these parameters). The chemical modulation of impedance can be frequency dependent. In that case the nominal order of this array increases. The principles and advantages of chemical modulation of WF as a sensing principle have been recently reviewed [3] and will not be repeated here. The purpose of this article is to explain the design of this new sensing platform and to present the results of the simple electrical and chemical tests as the illustration of the function of one sensing module. The development of different sensing layers, their deposition, the final packaging and performance of the multisensing array are in progress and will be reported in the near future in a separate publication.

2. Design and Fabrication of the Array

Chemical microsensor arrays are usually implemented in silicon. Clearly, the capability to fabricate a very large number of dedicated sensing elements exceeds the ability to create the number of orthogonal chemically sensitive layers by several orders of magnitude. Also, the ability to extract the raw outputs from the individual sensing elements presents a design and fabrication challenge. Thus, a fundamental design question needs to be asked: “how much raw data processing should be done on the same chip and how much should be done externally.” The answer to the question of how much “on-chip intelligence” should be designed is not simple [4]. In our opinion it depends on the information acquisition needs defined by the intended application. Thus, we come a full circle to the realization that the design of the chemical microsensor array and the selection of the sensing layers depends on the fully justified application. The design of individual chemical sensors for the sake of curiosity only is rarely an acceptable exercise.

The GT01 platform consists of two separately fabricated chips: the chemical sensing chip (CSC) and the electronic service chip (ESC). They perform two specific functions. The CSC houses the...
eight sensing modules which are separated from each other by a thick, photolithographically defined layer (Fig. 1). Its primary function is to define the areas into which the selective layers are deposited. The smallest feature size on the CSC is 20 \textmu m. Because the CSC occasionally comes into contact with harsh chemical environment the materials used for its fabrication must be chemically resistant, i.e., a high temperature silicon nitride as the passivation layer and a noble metal, e.g., gold, as the metallization layer. On the other hand, the ESC houses the electronic circuits used to extract the raw signals from the eight modules.

The ESC was laid out to a 1.25 \textmu m design rule and is fabricated by a standard CMOS process with aluminum metallization. The two chips are joined together by a flip-chip bonding with the ESC upside down on top of the bottom chip and the final encapsulation is done by a chemically resistant high grade encapsulant. This approach achieves several objectives:

- the preparation of the two chips requiring two different fabrication processes is separated,
- the packaging is greatly simplified because the critical electronic circuitry on the ESC is not exposed,
- the number of external wirebonds is minimized.

These attributes are somewhat less important for the gas sensing application but are critical for modification of chemically sensitive layers when the package is exposed to solutions of electrolytes. To our knowledge flip-chip bonding has not been used in fabrication of chemical sensors before. The idea is simple: two chips designed to perform two different functions (here the CSC and ESC) are fabricated by a separate process but the bonding pads (the points through which these two chips communicate electrically) are mirror images of each other. In the "flip-chip" process the bottom chip is kept in the wafer form while the top chip is diced (e.g., cut into individual dies). The top chip is carefully aligned face-to-face on top of the bottom chip (wafer) and the bonding is performed between the two sets of the bonding pads. This step always requires some heating and therefore the selection of the type of bonding process is also critical. In our case the CSC utilizes various polymers that preclude the use of high temperature solders. The wetting characteristics of low temperature solders for the bonding pad connections and their melting temperatures also limit their use in this application. A new procedure involving thermally curable, photolithographically processable conducting polymers is used instead [5]. In this procedure a 100 \textmu m high semi-cured "bumps" of conducting polymer are photolithographically defined on the ESC while it is still in the wafer form. After dicing the "bumped" ECS chips are flip-chip bonded to the CSC containing wafer as described above.

The circuit diagram implemented in each of the eight sensing modules of the GT01 package is shown in Figure 2. It employs a chemically sensitive field-effect transistor connected in a source-follower configuration. The sensing layer (typically a conducting polymer) serves as the gate. It is connected by two gold strips that have a dual purpose: they serve as the gate contacts (GATE A and B) and as the pair of electrodes for measurement of impedance of the gate material. This section of the circuit is implemented on each of the eight sensing modules located on the ESC (see box "CHEMFET & IMPEDANCE" in Fig. 2). The analog and the multiplexing circuitry are located on the ESC.

A constant current, \( I_{\text{in}} \), is applied to the CHEMFET as a bias signal. It is mirrored on-chip by cascade current mirrors 1 and 2 [6]. The signals \( E, S_n, I_n \), and \( V_{\text{in}} \) are applied to the GT01 from the external support circuitry. Each sensing module is selected by a signal, \( S_n \), that is generated by a 3-to-8 decoder that asserts only one of the 8 select lines. If the module is selected its output voltage is connected to the output lead of the GT01 package facilitating the measurement of the change in the WF of the CHEMFET gate. In combination with \( S_n \), an external enable signal \( E \) connected to the AND gate determines whether an input voltage \( V_{\text{in}} \) is connected to the other gate terminal of the CHEMFET. When \( E \) is applied, \( V_{\text{in}} \) is connected and the impedance between gate electrodes A and B can also be measured. In contrast, disabling \( E \) helps to minimize the adverse effects of the impedance measurements on the WF measurements.
The circuitry of the GT01 platform was simulated using H-Spice. The first ESC was fabricated by Hewlett Packard through the MOSIS prototyping service. The overall size of the ESC is 2863 \( \mu \text{m} \) \( \times \) 1076 \( \mu \text{m} \) and it has total of 27 aluminium (100 \( \times \) 100 \( \mu \text{m} \)) bonding pads. The CSC with \( n \)-channel CHEMFETs was fabricated in-house at the Georgia Tech Microelectronics Research Center with gold metallization and high temperature silicon nitride as the top passivation layer. The bonding pads of the CSC are a mirror image of the bonding pads on the ESC allowing flip-chip bonding of the two units. As the chemically sensitive layer is cast from a solvent into the 25 \( \mu \text{m} \) deep well, thicker edges along the walls of the well can develop in the selective layer (Fig. 3). Although this nonuniformity would have no effect on the WF measurement, it would adversely affect the measurement of impedance. For this reason, an impedance measuring area is defined by a thick SiO\(_2\) mask. This mask does not overlay the gate insulator area of the CHEMFET in order to allow the threshold voltage modulation along the entire width of the transistor channel. Thus, the opening in the SiO\(_2\) mask has the shape of a “cross”. For details about casting chemically sensitive layers by automated fluid microdispensing technique, see [7].

Packaging of the GT01 presents several challenges. First, the depth of the well into which the membrane is cast should be at least 25 \( \mu \text{m} \) to accommodate chemically sensitive layers of different conductivities. In our first approach, we have used a 25 \( \mu \text{m} \) thick photolithographically patterned polyimide (Ultradel 1414, AMOCO).

3. Testing of the Overall Package

The chemically sensitive layers based on polyaniline (PANI) for the detection of analytes in the gas phase have been deposited in the eight wells by casting of PANI dissolved in formic acid [8]. Since the PANI forms the gate conducting plate, testing of the transistor characteristics can be performed only after its deposition. \( n \)-Channel CHEMFETs with conjugated conducting polymers as gate materials are expected to have a positive threshold voltage. This is because the WF of most conducting polymers is considerably higher than that of p-type silicon [9]. As a result, in the source-follower configuration where the gate voltage (\( V_G \)) equals the drain voltage (\( V_D \)), the enhancement mode CHEMFETs will inherently operate in the saturated drain current region. Consequently, the sensor response will be in the first-order approximation independent of the \( I_D \) and \( V_{DS} \) and the relation between the analyte concentration and the measured WF will be predictable [3]. The CHEMFET characteristics are shown in Figure 4.

In the source-follower configuration the drain voltage/drain current and gate voltage/drain current curves follow the simple drain current equation only for \( V_G - V_T < V_D \), i.e., for enhancement mode transistors:

\[
I_D = \frac{\mu_wc}{2L} (V_G - V_T)^2
\]

Where \( \mu_c \) is the electron mobility in silicon, \( V_G \) is the applied gate voltage, \( V_T \) is the threshold voltage, \( V_D \) is the drain-to-
source voltage, $C$ is the gate capacitance per unit area, and $W$ and $L$ are the width and the length of the transistor channel, respectively. The plot of $(I_D)^{1/2}$ against $V_G$ is thus a straight line with $V_T$ being the intercept (Fig. 4).

The advantages of measuring both work function and bulk resistance changes on the same layers have been shown [10]. However, in that case, the sensor array was assembled from discrete sensors. In contrast, the GT01 can provide the same benefits as all sensors integrated on a single chip.

The full chemical evaluation of the responses of the GT01 chip coated with different chemically sensitive layers is in progress and will be reported separately. As the preliminary illustration of the performance, work function and impedance responses of the the GT01 chip with a PANI layer to ammonia are shown in Figure 5.

### 4. Conclusions

The design and fabrication of a first-order 8-sensor array for gas sensing has been accomplished. Work function and impedance responses measured on the one element have been demonstrated by exposing the devices with polyaniline sensing layer to ammonia. The testing was done in only a relatively narrow range of concentrations in order to demonstrate the functionality of the new design. A more comprehensive results of ammonia response of a dual-gate FET have been published previously [11]. The flip-chip bonding relaxes the conflict frequently encountered between fabrication requirements for the chemical and data processing electronics. The overall number of required wirebonds has been reduced from 27 to 10, making the fabrication economically feasible and more reliable.

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6. References


